



Breathability and Selectivity of Selected Materials for Protective Clothing

by Eugene Napadensky and Yossef A. Elabd

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14. ABSTRACT A series of materials was evaluated for its potential use as breathable (high water transport) barriers (low chemical agent transport) for military and civilian protective clothing apparel. The vapor transmission rates and effective permeabilities of water and dimethyl methyl phosphonate (DMMP) simulant for chemical agent (Sarin) through commercial and experimental polymeric membranes were measured using a modified American Society for Testing and Materials Method E-96-95. A variety of different materials from different companies was examined; unfortunately, some cannot be named due to proprietary restrictions. Materials examined included expanded PTFE, Nafion 117, sulfonated polystyrene-polyethylene-ran-polybutylene-polystyrene, sulfonated polystyrene-polyisobutylene-polystyrene, as well as four experimental and commercial membranes (A, B, C, and D). Results demonstrated that laminated membrane A had the highest breathability (effective permeability of water vapor); however, an experimental laminated material C possessed the highest selectivity with sufficient breathability, where selectivity was defined as the ratio of water permeability to DMMP permeability. Based on the results from this study, material C presented the greatest potential for a breathable barrier material for a chemical protective clothing application.					
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1. Introduction

Chemical and biological protective clothing technology remains a high priority among people responsible for the safety of U.S. military personnel. Not just from the obvious point of exposure to chemical or biological agents abroad and at home, but also from another, less often considered, but still important safety factor: breathability. Until recently the main goal in the development of chemical and biological protective clothing was to maximize protection. In the past, one of the more commonly used materials for this application was butyl rubber working on principle of total blockage. Butyl rubber is an effective barrier to most harmful agents at certain thicknesses, but it has certain disadvantages. Wearing garments manufactured from this material in combat or in other situations which involves physical activity is not practical because the lack of breathability of this material causes heat fatigue and exhaustion (1). A different approach for protection against chemical and biological threats is sorption materials. Protective suits such as the Military Oriented Protective Posture (MOPP) suit and the Joint Service Lightweight Integrated Suit Technology (JSLIST) work on principle of capturing toxic materials with fillers like activated carbon. JSLIST is more breathable, but it does not provide as much protection as butyl rubber. It is also relatively heavy, bulky, and working in the suit is limited to ~45 min/hr (2).

A completely different approach to the problem of chemical and biological protection is the concept of semi-permeable membranes. That would be materials that allow passage of moisture (perspiration), but block harmful molecules and organisms. Materials like that will offer protection in the hostile environment without causing significant heat fatigue and exhaustion. A number of such semi-permeable membranes are being developed in industry and at the U.S. Army Research Laboratory (ARL).

The scope of this work was to examine a series of semi-permeable membranes in order to evaluate their potential usefulness as a breathable, yet agent-resistant, material for military and civilian clothing. This study examines the permeation of water vapor and the permeation of dimethylmethylphosphonate (DMMP) vapor through a variety of materials to determine the best candidates that are both breathable and protective. DMMP is chemically similar to the nerve agent Sarin (GB) and is often used to imitate the behavior of chemical agents when the usage of actual agents is cost prohibitive due to safety concerns. Materials examined in this study include some commercial materials as well as a number of experimental materials under development both at ARL and other laboratories. Due to proprietary restriction and other legal considerations, it was decided that four materials cannot be named and will be referred to as materials A, B, C, and D with manufacturers referred to by the same letter.

2. Experimental

2.1 Materials

Materials evaluated in this study are listed in table 1. Sulfonated poly(styrene-isobutylene-styrene) (S-SIBS) at both 58 and 85 mole-percent sulfonation, referred to as S-SIBS-58 and S-SIBS-85 in table 1, respectively, was sulfonated at ARL and the details of this procedure can be found elsewhere (3). Mole-percent sulfonation is defined as moles of sulfonic acid per moles of styrene repeat unit and was determined by elemental analysis. The unsulfonated polymer was provided by Kuraray Co., Ltd., Tsukuba Research Laboratories, with the reported properties: 30.84 weight-percent styrene, 0.95 specific gravity, $M_w = 71,920$ g/mol, $M_n = 48,850$ g/mol, and polydispersity index (PDI) = 1.47. Sulfonated poly(styrene-ethylene-*ran*-butylene-styrene) (S-SEBS) at both 59 and 93 mole-percent sulfonation, referred to as S-SEBS-59 and S-SEBS-93 in table 1, respectively, was sulfonated at ARL and the general details of this procedure can be found elsewhere (4, 5). Unsulfonated poly(styrene-ethylene-*ran*-butylene-styrene) was provided by Kraton Polymers. S-SEBS-50 is a poly(styrene-ethylene-*ran*-butylene-styrene) sulfonated to 45%–55% that was purchased from Sigma-Aldrich as a solution and cast into films at Natick Soldier Command Center. Other materials in this study included an expanded PTFE membrane, Nafion 117* (DuPont), and another four materials A, B, C, and D from corresponding companies. In addition, other chemicals used in these experiments were as follows: water (J. T. Baker, high-performance liquid chromatography [HPLC] grade), dimethylmethylphosphonate (Lancaster Synthesis, 97%), Drierite† (Anhydrous Calcium Sulfate, 97%, W. A. Hammond Drierite Co.), toluene (EM Science, 99.5%), and hexanol (J. T. Baker).

Table 1. List of evaluated materials and their thickness.

Material Name	Source and Manufacturer	Sample Thickness (μm)
S-SIBS-58	ARL	103–159
S-SIBS-85	ARL	89–115
S-SEBS-59	ARL	129–188
S-SEBS-93	ARL	111–174
S-SEBS-50	Aldrich	119–204
A (cloth laminate)	A	264–372
Expanded PTFE	Natick SCC	43–48
B	B	13–16
C	C	179–187
D	D	39–42
Nafion 117	DuPont	181–186

* Nafion 117 is a registered trademark of DuPont.

† Drierite is a registered trademark of W. A. Hammond Drierite Company.

2.2 Permeation Equipment

Equipment used for vapor permeation experiments consisted of a temperature-controlled oven with a nitrogen gas sweep passing through a Drierite-packed column. Aluminum trays with Drierite were also placed inside the oven to maintain a low-relative humidity (~10%). In addition, an analytical balance (precision = ± 0.0001 g) was used to measure weight loss and a digital micrometer was used to measure the thickness of each membrane (precision = ± 1.2 μm).

2.3 Membrane Preparation

Membranes developed at ARL (S-SIBS-58, S-SIBS-85, S-SEBS-59, S-SEBS- 93) were prepared by solution casting in Teflon* petri dishes at 2.5% w/v of polymer in 85/15 w/w toluene and hexanol. All membranes were cast in open air for 2 weeks, then annealed in a vacuum oven at 50 °C for 1 or 2 weeks to remove residual solvent. All other membranes were used as received.

2.4 Vapor Permeation Procedure

2.4.1 Cap Loading

Twenty-mL vials, with open-top caps (VWR) and Teflon-lined septa with a 14-mm hole cut in the center of the septa to match the hole in the cap were used (figure 1). Membranes were cut into circles with a 22-mm diameter, the size of the original septa. The thickness of each individual membrane was measured with a digital micrometer. Membranes were placed inside the cap with the cored septa placed behind to provide an air tight seal.

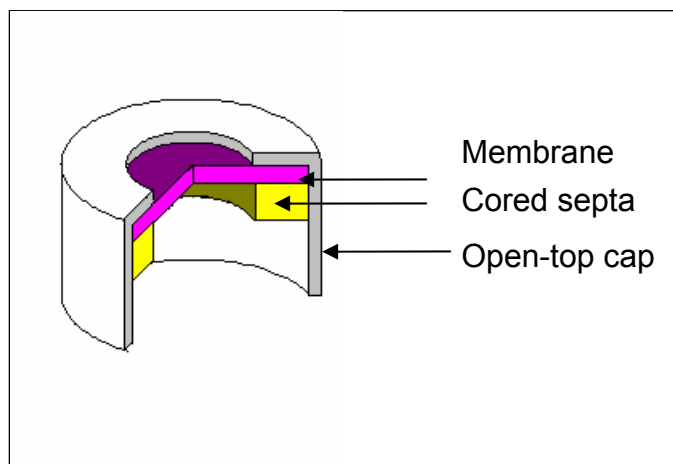


Figure 1. Schematic representing a cutaway of the cap-membrane assembly showing an open-top cap, membrane, and cored septa.

* Teflon is a registered trademark of DuPont.

For samples A and C, the polymeric membranes which are laminated onto cloth were loaded into the cap assembly with the cloth side positioned toward the permeant for DMMP vapor permeation and positioned away from the permeant for water vapor permeation.

2.4.2 Vapor Permeation

Vapor permeation experiments were conducted based on the American Society for Testing and Materials (ASTM) E 96-95 (Standard Test Methods for Vapor Transmission of Materials) procedure (6). An oven, with nitrogen gas purge passed through a desiccant trap, was stabilized at 35 °C and 10% relative humidity (RH). Vials were filled with 10–15 mL of liquid (water or DMMP) and placed in oven to equilibrate for about 24 hr. Vials were removed, regular caps were replaced with membrane lined caps shown in figure 1. The total assembly weight was recorded, and the vials were placed back into the oven to initiate the experiments. The complete membrane and vial assembly is shown in figure 2.

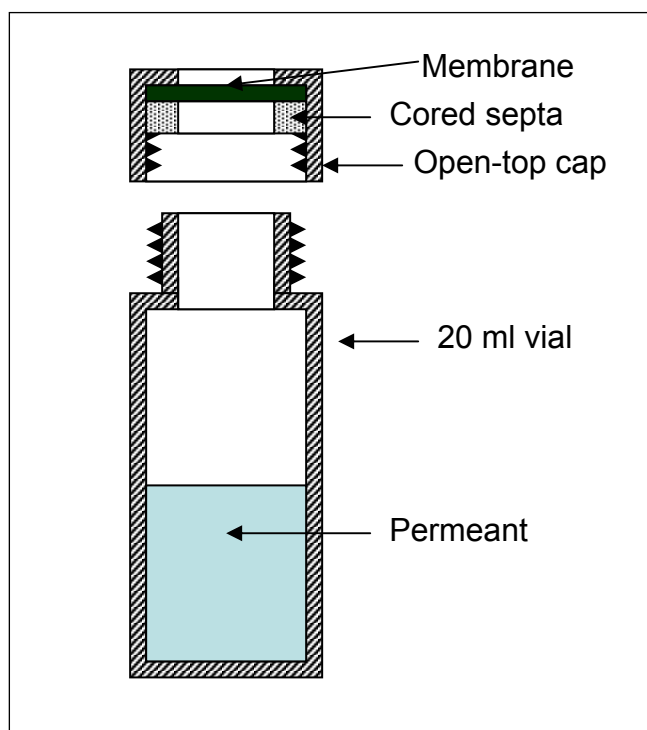


Figure 2. Complete vial assembly used for vapor permeation experiments.

Vial weight, temperature, and humidity inside the oven were recorded every 24 hr until a sufficient amount of data was collected to determine constant rate of weight loss for each vial (steady-state). RH remained constant at 10% throughout the duration of the experiment. When using water as a permeant, the conditions were 100% RH on one side of the membrane (inside the vial) and 10% RH on the other side (outside the vial). The concentration gradient provided the driving force for vapor transport. Experiments with DMMP vapor were at 100% DMMP

saturation (6.77-mm Hg) on one side of the membrane (inside the vial) and 0% DMMP on the other side or outside the vial.

Three experiments were conducted for each membrane, and the values calculated for each membrane are the average and standard deviation of those experiments.

3. Results and Discussion

3.1 Theory

Vapor transfer rate (VTR) is defined as steady state VTR per unit area and can be expressed as follows:

$$\text{VTR} = \frac{G}{(t * A)}, \quad (1)$$

where G is weight of penetrant, t is time, and A is cross-sectional area. For this experiment, the cross-sectional area is 0.000154 m² because the diameter is constant at 14 mm. G/t can be regressed from the steady state portion of the weight loss data vs. time, as shown in figure 3.

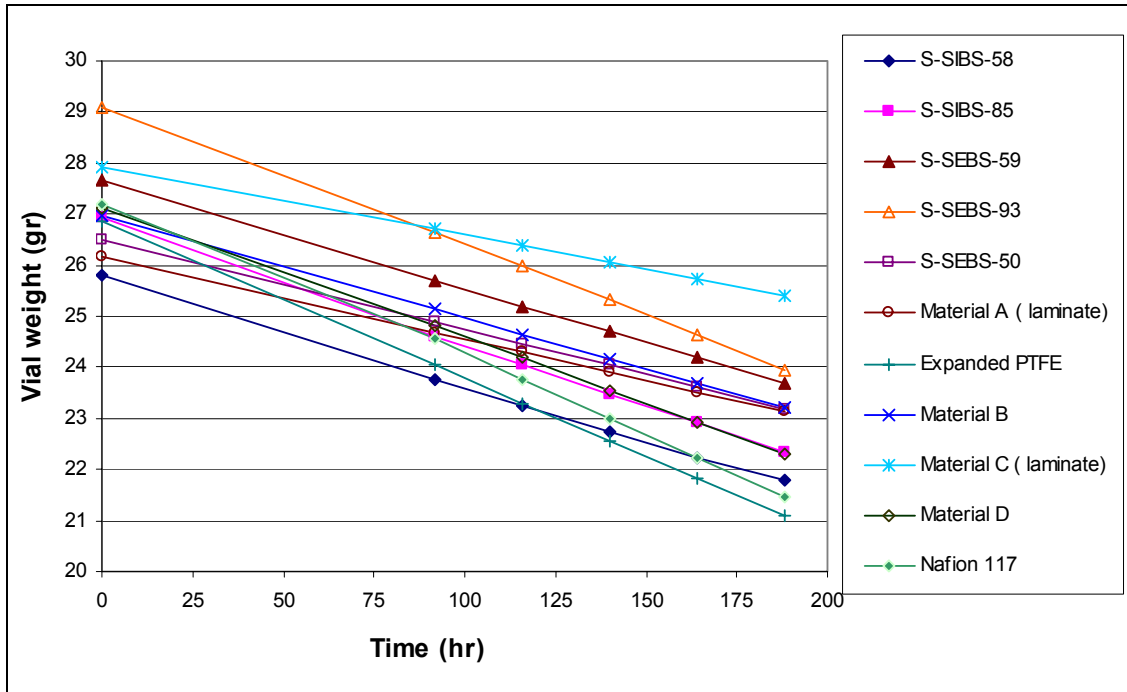


Figure 3. Weight loss during water vapor permeation experiments.

After calculating G/t from the data, VTR can be obtained using equation 1.

VTR provides transport rate for a given penetrant through a membrane. However, VTR does not account for the thickness of the membrane effectively. More specifically, VTR will have different values for the same material at different thicknesses. To accurately compare materials independent of the processing thickness, an effective permeability must be calculated. Effective permeability (P_{eff}) can be expressed as:

$$P_{\text{eff}} = \frac{L * \text{VTR}}{S * (P_1 - P_2)}, \quad (2)$$

where S is the saturation vapor pressure at the test temperature (mm Hg), P_1 is the partial pressure or relative humidity on the challenge side, P_2 is partial pressure on the exit side, and L is the sample thickness (m). For laminated samples (materials A and C), L was taken to be thickness of the full system, cloth and all the layers of lamination. Saturation vapor pressure for water at 35 °C is 41.175-mm Hg (7) and for DMMP at 35 °C, it is 6.77-mm Hg. The DMMP vapor pressure was determined by interpolating from known values at 25 and 65 °C (8).

3.2 VTR and P_{eff}

Figures 4 and 5 show water VTR and P_{eff} results for all of the materials studied. Since breathability is one the key goals, it is tempting to concentrate on materials with a high water VTR. However, since VTR values do not account for sample thickness, a high VTR value does not necessarily signify sufficient breathability. Good examples include expanded PTFE, material B, and material D. All three samples have high VTRs (figure 4), but when their thicknesses are taken into consideration, their effective permeability, P_{eff} , (breathability) is significantly lower than other materials (figure 5).

Figures 6 and 7 show VTR and P_{eff} results for DMMP, respectively. DMMP simulates the nerve agent Sarin, and the optimum material is one that minimizes DMMP transfer for good barrier properties against chemical agents. Figure 7 shows that materials B, C, and D have the lowest values of DMMP P_{eff} and would provide the best protection against chemical agents.

3.3 Selectivity

The goal for chemical and biological defense is to identify materials that will provide sufficiently high water transport (breathability) to reduce heat fatigue, and sufficiently low DMMP transport to reduce personnel risk from harmful agents. The performance of these materials can be examined by plotting $P_{\text{eff}}(\text{DMMP})$ vs. $P_{\text{eff}}(\text{WATER})$, as shown in figure 8.

An actual numerical value of ratio of effective permeabilities is defined as selectivity (α):

$$\alpha = \frac{P_{\text{eff}}(\text{WATER})}{P_{\text{eff}}(\text{DMMP})}, \quad (3)$$

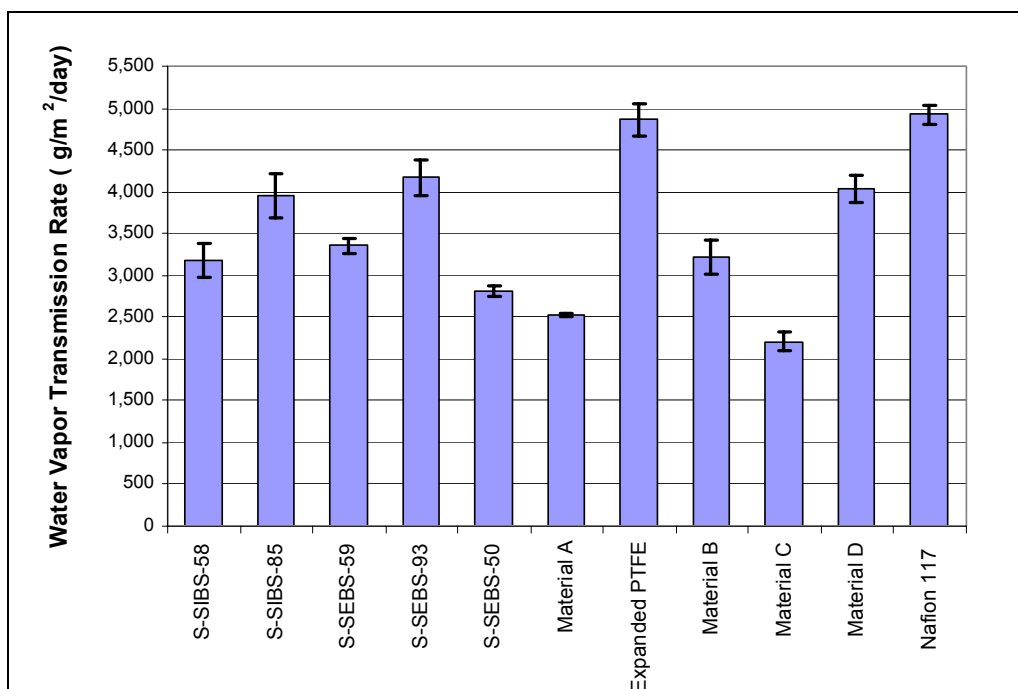


Figure 4. Water vapor transmission rate.

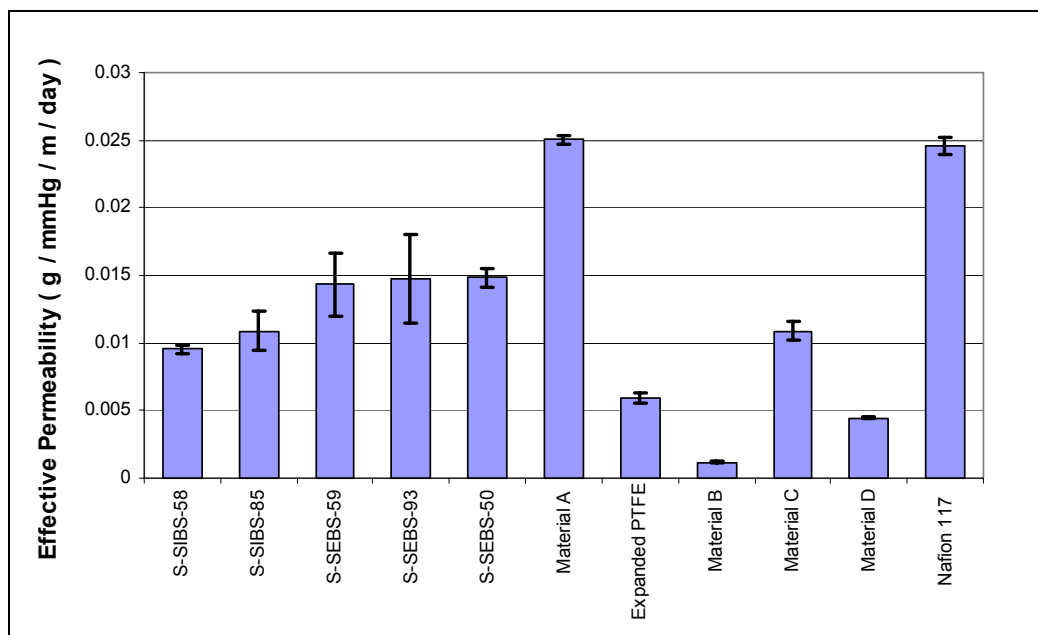


Figure 5. Water-effective permeability results.

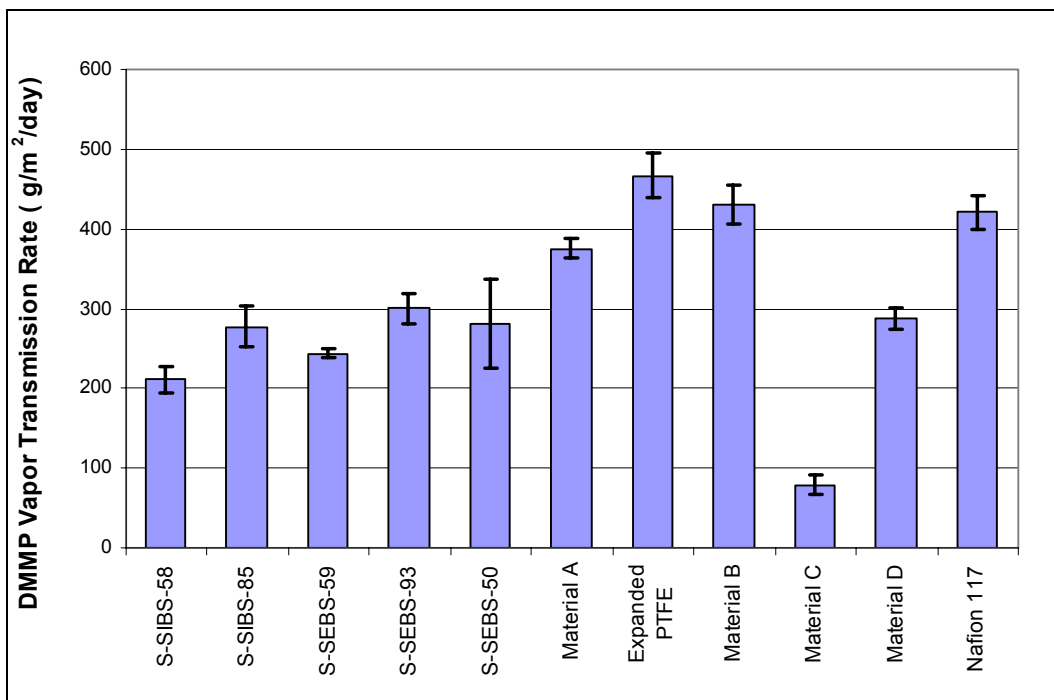


Figure 6. DMMP vapor transmission rate.

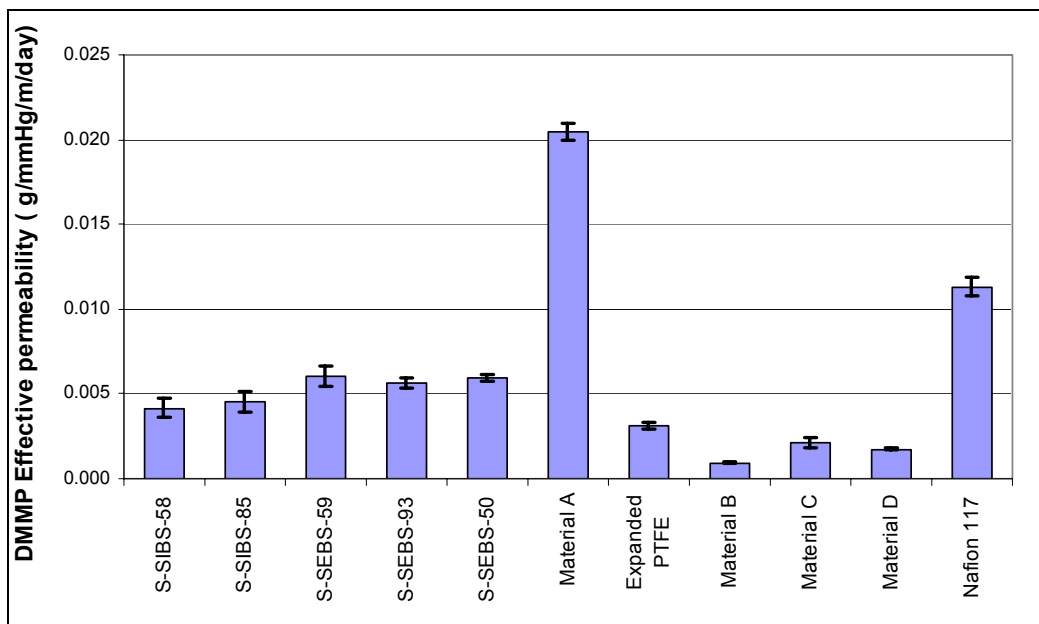


Figure 7. DMMP-effective permeability results.

where a high value would be desired for a breathable protective material. This is the slope of the trend line shown in figure 8. The upper left corner of this plot is where an ideal material for breathable protective clothing would be located. This corresponds to a very high water transport and simultaneously very low agent transport or a high selectivity.

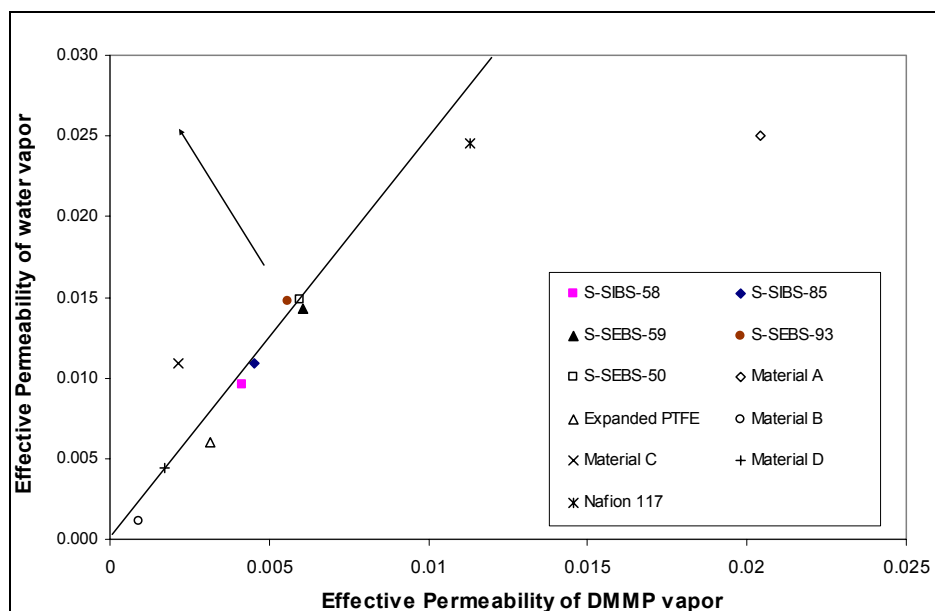


Figure 8. Relative performance of materials based on effective permeability.

Selectivity values for materials examined in these experiments are shown in figure 9. Selectivity does not account for actual amount of penetrant going through membrane. A material that has high values for effective permeation for both penetrants may have the same selectivity as

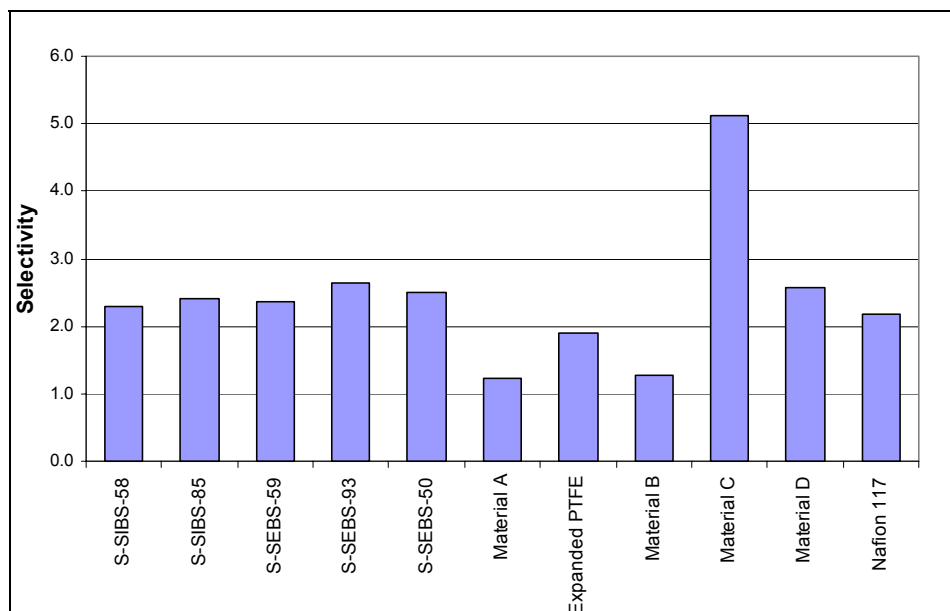


Figure 9. Selectivity of materials.

a material that has low values for both penetrants. Therefore, generally selectivity cannot be used as a final criteria, but rather should be used in conjunction with P_{eff} (water vapor). However, in this study, the water vapor transport for all materials (figures 4 and 5) provide sufficient breathability, and therefore, materials with the highest selectivities have the desired properties for breathable protective clothing (9). Figure 9 shows that material C has a selectivity that is more than double compared to the other materials in this study.

4. Conclusion

In this study, the water vapor transmission rate, effective permeability, and selectivity of water vapor and DMMP vapor (simulant for chemical warfare agent) were measured for a variety of commercial and experimental candidate materials for evaluation as a breathable barrier for protective clothing. Material C has sufficient water vapor permeability (breathability) and a selectivity (5.12) that is more than double compared to the rest of the materials evaluated. If high selectivity is used as an indicator, then out of the materials evaluated in this study, this one has a good potential for use as a breathable barrier for protective clothing. Of course, there are still other factors that need to be considered, such as durability, price, weight, process ability, and other physical properties. Over all, this preliminary study indicates that further research on the synthesis and testing of new semi-permeable materials may produce more efficient breathable barriers not only protective clothing for the military, but also for apparel for a variety of other applications.

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Appendix A: Raw Data

Table A-1. Sample thickness and weight of vial assembly at a given time. Permeant is water. Conditions: 35 °C, 100% RH side A, and 10% RH side B.

Water								
Exposure Time (hr) ->		Thickness (μm)	0	92	116	140	164	188
S-SIBS-58	A1	109	25.81	23.7504	23.2428	22.7492	22.2435	21.7727
	A2	103	25.957	23.9543	23.4171	22.8883	22.3772	21.8976
	A3	124	30.95	28.0352	27.5758	27.1353	26.6892	26.1969
S-SIBS-85	B1	102	26.935	24.5824	24.0451	23.4724	22.9038	22.3436
	B2	115	27.867	25.4099	24.8006	24.1878	23.5615	22.9138
	B3	89	26.175	23.8069	23.1506	22.5163	21.8649	21.2498
S-SEBS-59	C1	180	27.649	25.7069	25.1955	24.6922	24.1822	23.6915
	C2	165	29.961	27.806	27.2735	26.7396	26.2033	25.6764
	C3	129	28.664	26.6218	26.1177	25.6013	25.0809	24.5677
S-SEBS-93	D1	111	29.091	26.6406	25.9849	25.3156	24.6322	23.94
	D2	112	26.898	24.5245	23.8562	23.2132	22.5651	21.9528
	D3	174	27.6	25.4118	24.8014	24.1948	23.5794	22.9894
S-SEBS-50	E1	189	26.503	24.8842	24.4613	24.036	23.6085	23.1898
	E2	194	26.608	24.9407	24.5096	24.0696	23.622	23.1756
	E3	204	27.161	25.5268	25.1007	24.6711	24.236	23.7949
Material A (cloth laminate)	F1	364	26.183	24.6887	24.2904	23.906	23.5152	23.1394
	F2	364	26.815	25.2925	24.8936	24.5012	24.1052	23.7171
	F3	372	28.211	26.703	26.3152	25.9268	25.5368	25.1536
Expanded PTFE	G1	48	26.865	24.0392	23.3022	22.5587	21.8149	21.0809
	G2	43	27.402	24.6028	23.8866	23.1629	22.4373	21.702
	G3	45	26.592	23.5748	22.7577	21.9776	21.1942	20.441
Material B	H1	13	26.956	25.1371	24.6523	24.1764	23.6977	23.2253
	H2	15	26.839	25.0188	24.5366	24.0597	23.5797	23.1042
	H3	13	26.614	24.7743	24.2444	23.7175	23.1852	22.6532
Material C (on cloth)	I1	179	27.936	26.6991	26.3784	26.0559	25.7303	25.3981
	I2	184	27.709	26.4423	26.0724	25.718	25.3606	25.0048
	I3	187	26.977	25.7415	25.3991	25.0632	24.7267	24.3935
Material D	J1	41	27.103	24.814	24.1824	23.5555	22.9306	22.3022
	J2	42	26.572	24.2886	23.6986	23.1076	22.5099	21.9051
	J3	40	27.657	25.2216	24.613	23.9864	23.3307	22.6518
Nafion 117	K1	186	27.177	24.5714	23.7643	22.9908	22.2352	21.4656
	K2	183	26.973	24.4624	23.695	22.9318	22.1877	21.4259
	K3	185	29.103	26.7624	26.0163	25.2733	24.5473	23.8026

Table A-2. Sample thickness and weight of vial assembly at a given time. Permeant is DMMP. Conditions: 35 °C, 100% saturation side A, and 0% saturation side B. RH = 10% both sides.

DMMP									
Exposure Time (hr) ->		Thickness (μm)	0	90	114	138	162	186	258
S-SIBS-58	A1	114	32.5163	32.4117	32.3768	32.3416	32.3083	32.2731	32.163
	A2	130	33.1398	33.0479	33.0171	32.9872	32.9583	32.9274	32.8347
	A3	159	32.9086	32.822	32.7908	32.7593	32.7295	32.6975	32.6007
S-SIBS-85	B1	115	32.7154	32.5869	32.5411	32.4952	32.4505	32.4038	32.255
	B2	107	32.2355	32.1049	32.0636	32.0248	31.986	31.9456	31.8215
	B3	108	34.5352	34.4027	34.3624	34.323	34.2842	34.2436	34.1181
S-SEBS-59	C1	165	32.7134	32.59	32.5537	32.5175	32.4824	32.4456	32.3322
	C2	188	33.5829	33.4583	33.4214	33.3851	33.3495	33.3123	33.1975
	C3	152	33.1566	33.0194	32.9802	32.9422	32.9055	32.8673	32.748
S-SEBS-93	D1	115	32.2193	32.0597	32.0101	31.9621	31.9147	31.8659	31.7179
	D2	131	32.9339	32.7754	32.7288	32.6825	32.6374	32.59	32.4449
	D3	133	33.1807	33.034	32.9919	32.9501	32.9086	32.8653	32.7321
S-SEBS-50	E1	144	32.6787	32.5383	32.4972	32.457	32.4175	32.3761	32.2486
	E2	119	33.4826	33.3235	33.2686	33.2148	33.1637	33.1118	32.9546
	E3	180	32.5887	32.4776	32.4425	32.4075	32.3732	32.337	32.228
Material A (cloth laminate)	F1	365	33.2753	33.0494	32.9914	32.9329	32.8754	32.8159	32.6303
	F2	368	33.3663	33.1483	33.092	33.0363	32.9812	32.9242	32.7448
	F3	371	32.9934	32.7797	32.7243	32.6706	32.6166	32.5611	32.3855
Expanded PTFE	G1	45	32.8536	32.5868	32.5196	32.4538	32.388	32.32	32.108
	G2	46	33.8087	33.5325	33.4637	33.396	33.3277	33.2573	33.0381
	G3	45	32.9828	32.6531	32.5782	32.5036	32.4297	32.3537	32.115
Material B	H1	13	33.9629	33.6801	33.6087	33.5396	33.472	33.4027	33.1841
	H2	16	33.0792	32.8333	32.7699	32.7085	32.6479	32.5856	32.3895
	H3	15	32.9225	32.6739	32.6076	32.5446	32.4814	32.417	32.2177
Material C (on cloth)	I1	186	33.1451	33.1308	33.1214	33.1104	33.1019	33.0923	33.0611
	I2	182	32.9224	32.8995	32.885	32.87	32.8578	32.8447	32.8026
	I3	183	32.7894	32.7692	32.7582	32.7444	32.7328	32.72	32.6809
Material D	J1	39	32.245	32.0685	32.022	31.9762	31.9306	31.8844	31.7422
	J2	42	32.7597	32.5995	32.5552	32.5125	32.47	32.4267	32.2918
	J3	41	32.7042	32.546	32.504	32.4625	32.4207	32.378	32.2468
Nafion 117	K1	181	32.7303	32.5144	32.4481	32.3811	32.3144	32.2466	32.0345
	K2	182	33.4008	33.1927	33.131	33.0695	33.0076	32.9442	32.749
	K3	182	32.7772	32.5757	32.5145	32.454	32.3927	32.3305	32.1383

Appendix B: Calculation Results

Table B-1. Calculation results.

	Water				DMMP				
	Water VTR		Effective Permeability		DMMP VTR		Effective Permeability		Selectivity
	g/m ² day	STDEV	(g/mmHg m day)		g/m ² day	STDEV	(g/mmHg m day)		
			Av	STDEV			Av	STDEV	
S-SIBS-58	3175	197.41	9.56E-03	3.16E-04	210.9	17.0	4.16E-03	5.67E-04	2.30
S-SIBS-85	3955	271.09	1.09E-02	1.47E-03	277.6	25.7	4.52E-03	5.96E-04	2.41
L-SEBS-59	3357	95.26	1.43E-02	2.38E-03	243.8	6.1	6.06E-03	5.84E-04	2.36
S-SEBS-93	4168	218.46	1.48E-02	3.28E-03	300.8	18.7	5.60E-03	3.05E-04	2.64
S-SEBS-50	2812	54.75	1.48E-02	7.09E-04	280.2	55.9	5.95E-03	2.41E-04	2.49
Material A (cloth laminate)	2531	23.82	2.50E-02	3.67E-04	375.7	11.8	2.04E-02	4.97E-04	1.22
Expanded PTFE	4864	194.73	5.97E-03	3.88E-04	467.1	28.6	3.13E-03	1.86E-04	1.91
Material B	3217	198.03	1.18E-03	9.74E-05	430.5	25.1	9.26E-04	4.08E-05	1.27
Material C (on cloth)	2203	110.61	1.09E-02	7.01E-04	78.6	12.8	2.13E-03	3.24E-04	5.12
Material D	4038	158.23	4.46E-03	7.61E-05	288.0	12.7	1.73E-03	5.43E-05	2.58
Nafion 117	4921	117.02	2.45E-02	6.28E-04	420.7	21.3	1.13E-02	5.41E-04	2.17

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Appendix C: Sample Description

- S-SIBS-58: Sulfonated (58%) polystyrene-polyisobutylene-polystyrene. Solvent cast. Yellowish elastomeric film. Sulfonated and cast at the U.S. Army Research Laboratory (ARL). Sample thickness: 103–59 μm .
- S-SIBS-85: Sulfonated (85%) polystyrene-polyisobutylene-polystyrene. Solvent cast. Yellowish elastomeric film. Sulfonated and cast at ARL. Sample thickness: 89–115 μm .
- S-SEBS-59: Sulfonated (59%) polystyrene-polyethylene-ran-polybutylene-polystyrene. Solvent cast. Yellowish elastomeric film. Sulfonated and cast at ARL. Sample thickness: 129–188 μm .
- S-SEBS-93: Sulfonated (93%) polystyrene-polyethylene-ran-polybutylene-polystyrene. Solvent cast. Yellowish elastomeric film. Sulfonated and cast at ARL. Sample thickness: 111–174 μm .
- S-SEBS-50: Sulfonated (45%–55%) polystyrene-polyethylene-ran-polybutylene-polystyrene. Purchased from Aldrich, solvent cast at Natick SCC. Yellowish elastomeric film. Sample thickness: 119–204 μm .
- Material A: Three-layer laminated cloth, woodland camouflage on external side, and olive green on internal. Manufactured by company A, obtained from Natick SCC. Sample thickness (total): 264–372 μm .
- Expanded PTFE: Thin white film. Obtained from Natick SCC. Sample thickness: 43–48 μm .
- Material B: Whiteish, semi-transparent polyester/polyether membrane manufactured by company B. Obtained from Natick SCC. Sample thickness: 13–16 μm .
- Material C (cloth laminate): Experimental laminate manufactured by company C. Woodland camouflage cloth on external side and white polymeric film on internal. Sample thickness (total): 179–187 μm .
- Material D: Experimental membrane manufactured by company D. Yellowish elastomeric film. Sample thickness: 39–42 μm .
- Nafion 117: Perfluorinated membrane, hydrogen ion form. Yellowish elastomeric film. Manufactured by DuPont. Sample thickness: 181–186 μm .

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